

Micro Analytical Systems Department Technology– μ ChemLab™

Fact Sheet

Abstract

Sandia has developed a hand-held chemical analysis system. The instrument uses three microfabricated analysis stages. Stage one collects and concentrates the samples known as analytes. Stage two is a gas chromatography (GC) column used to separate the analytes. Stage three is an array of surface acoustic wave (SAW) sensors used to identify and quantify the separated analytes.

Introduction

In recent years, sensors have evolved from discrete devices (requiring separate electronics) to integrated devices, where sensors and operating electronics are co-located on a chip “smart sensors.” Recently chemical sensors have benefited from a parallel evolution in which micromachined MEMS structures capable of performing sample handling and chemical separations have been added to on-chip sensors and electronics. These *micro-analytical systems* exhibit phenomenal increases in discrimination (ability to distinguish between analytes) over sensors alone. These systems promise to revolutionize a number of fields, including food processing, health care, and chemical/biological weapon defense.

We have developed a “hybrid” system, known as μ ChemLab™, where chemical separation (using a short column for rapid analysis with less resolution) is performed with the addition of an array of chemical detectors [1]. This combination approach provides rapid and discriminating analyses. The μ ChemLab™ combines three cascaded stages; each realized using microfabricated components, (along with a miniature pump) for the analytical system (Fig. 1).

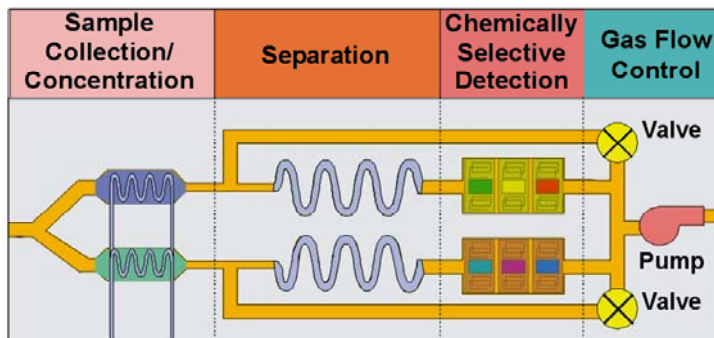


Fig. 1. Schematic for the μ ChemLab™ gas-phase chemical analysis system.



Collection/Concentration Stage

The first stage of the $\mu\text{ChemLab}^{\text{TM}}$ system is the preconcentrator unit. The preconcentrator unit is a sample collection/concentration stage and collects “select” analytes from an inlet gas sample stream and ejects them on command into the separation stage. Typical sample collection times from the inlet gas stream are 30 to 60 seconds. This stage consists of a thin silicon nitride membrane supporting a patterned metal film heating element (Fig. 2). The membrane is coated with a pre-selected film to selectively and reversibly absorb analytes of interest.

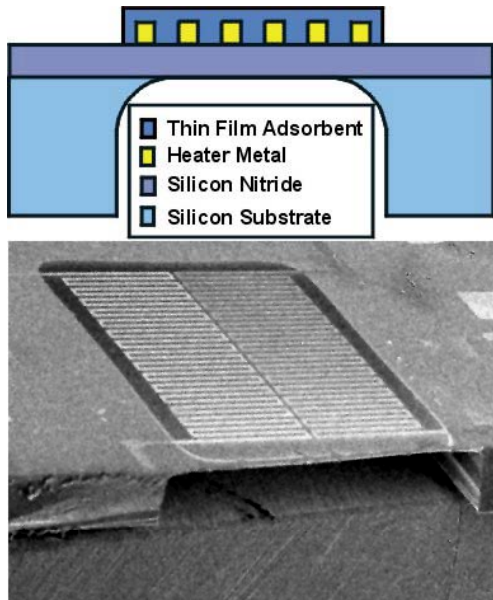


Fig. 2. Collection/concentration stage of the $\mu\text{ChemLab}^{\text{TM}}$ system, consisting of a membrane supporting a metal film heater and chemical trap.

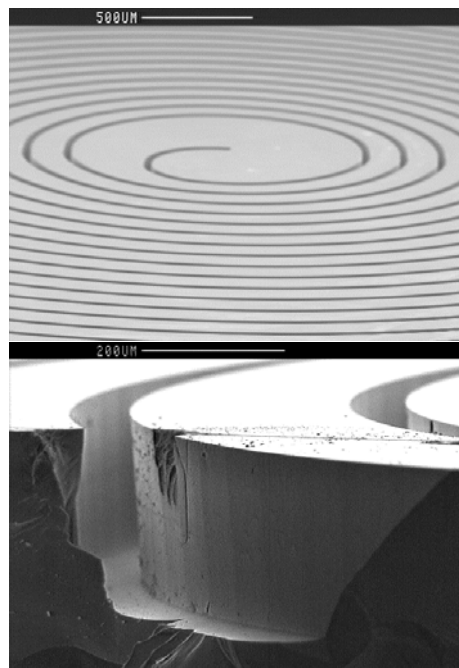


Fig. 3. Micromachined (open column) gas chromatograph used for separating analytes in the $\mu\text{ChemLab}^{\text{TM}}$ chemical analysis system.

Separation Stage

The second stage in the $\mu\text{ChemLab}^{\text{TM}}$ is the GC separation stage. This stage involves a high aspect ratio (depth-to-width) GC column that is fabricated using deep reactive ion etching into a silicon wafer (Fig. 3). The column is spiral shaped and occupies only 1 cm² of chip area and is typically about 1 m in length. The column cross-section is typically 50 – 100 μm wide by 400 μm deep. The high aspect ratio provides a large flow cross-section, while maintaining effective separation due to the small width. Typically air is used as the carrier gas (5 psi across the column) providing separations in 30 – 60 seconds.

The walls of the GC column can be coated with a stationary phase that selectively retains different analytes. The choice of the coating (stationary phase) used depends on the analytes of interest. Stationary phases range from the very nonpolar polydimethylsiloxane to the highly polar polyethylene glycol. Polar phases typically interact strongly with polar analytes, causing longer elution times and better separations; likewise, non-polar phases are best suited for non-polar analytes. The interactions of the analyte and the stationary phase mean that the analytes are repeatedly sorbed and desorbed as the sample flows through

the column. The analytes emerge from the column in distinct narrow bands based on the analyte's interaction with the column, giving a chromatogram that allows for the identification of each constituent.

Fig. 4 is a chromatogram showing the separation of benzene, toluene, the three xylene isomers, and octane. The separation is performed in only 30 seconds. The open channel GCs shown in Fig. 3 are effective for separating high boiling point chemicals (vapors of chemicals normally liquid at room temperature). For separating low boiling point analytes (normally gases at room temperature), columns packed with beads that are coated with a stationary phase are typically used (Fig. 5). Fig. 6 shows a chromatogram showing the separation of natural gas constituents using the packed column on a chip. Methane (C1), ethane (C2), propane (C3) and two isomers of butane (n-butane and isobutane, both C4) are discriminated.

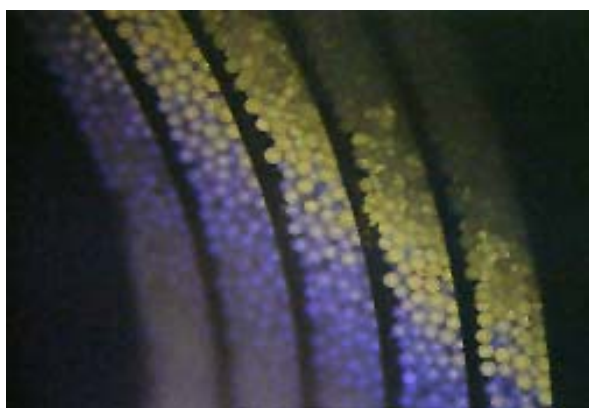


Fig. 5. Micromachined gas chromatograph column packed with beads, suitable for separation of low boiling point gases.

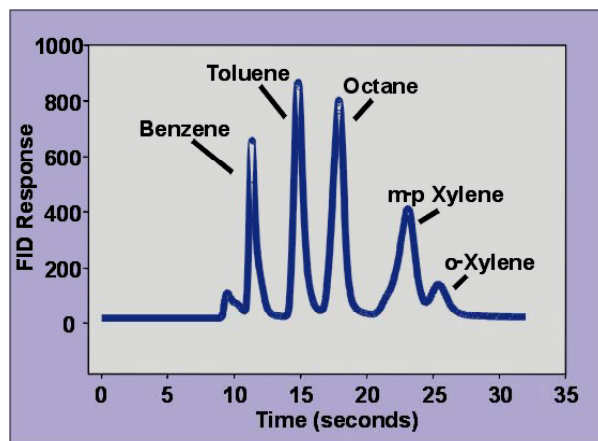


Fig. 4. Chromatogram obtained with an open channel GC column of the type shown in Fig. 6, showing separation of five hydrocarbons. Detection is performed using an FID detector

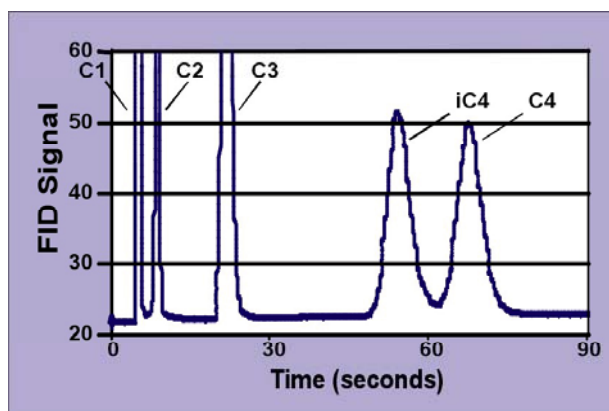


Fig. 6. Chromatogram obtained with a packed-column GC of the type shown in Fig. 5, showing separation of low boiling point gases. Detection is performed using an FID detector.

Chromatographic separations can be performed either isothermally or with temperature ramping (variable temperature). The latter provides some advantages in separating mixtures of analytes with widely varying boiling points. A heater and temperature probe are affixed to the back of the GC column and are used to control the temperature.

Detection Stage

The third stage of the $\mu\text{ChemLab}^{\text{TM}}$ system is the detection unit. The detection method uses acoustic wave propagation over coated surfaces. The detector consists of an array of surface acoustic wave (SAW) sensors (Fig. 7). Each SAW device consists of an input and output interdigital transducer patterned on a piezoelectric substrate. When the input transducer is excited at its synchronous frequency (typically 100MHz to 1 GHz) the input transducer generates a surface acoustic wave that propagates across the crystal and regenerates an electrical signal on the output transducer [2]. The surface wave has acoustic energy confined to within roughly one wavelength of the surface, making the wave extremely sensitive to accumulated surface mass.

To make the SAW device a chemical sensor, a sorbent film is deposited onto the SAW propagation path. As each analyte exits the GC column and passes across the SAW sensor, the coating momentarily absorbs the analyte. The resulting minute increase in surface mass causes a decrease in the SAW propagation velocity.

The SAW sensor array typically consists of three sensors with different sorptive coatings and an uncoated reference device (Fig. 7). The array thus provides several responses that can be used to differentiate analytes that co-elute from the GC column, providing an extra degree of discrimination over the temporal separation provided by the separation stage.

The electronics to operate the SAW array are built into an application specific integrated circuit (ASIC) that is attached to the SAW substrate to form a monolithic integrated sensor array (Fig. 7). The ASIC provides RF drive and phase detection circuitry for measuring the minute changes in SAW velocity arising from absorption of chemicals.

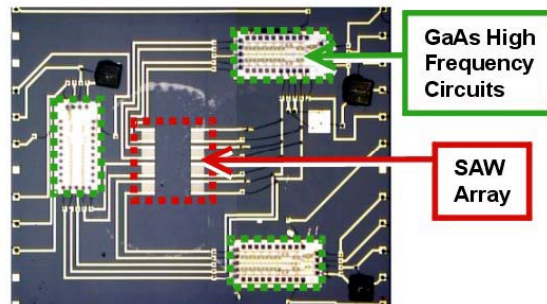


Fig 7. An array of surface acoustic wave (SAW) sensors used as a detector on the output of the gas chromatograph column in the $\mu\text{ChemLab}^{\text{TM}}$ system. An ASIC is attached to provide RF drive and phase detection for the SAW sensors.

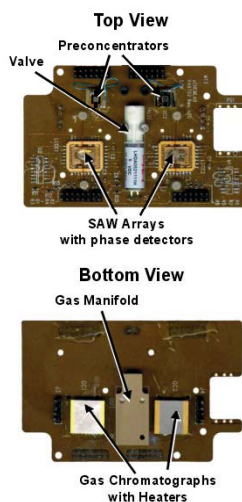


Fig. 8. Left: PC board with $\mu\text{ChemLab}^{\text{TM}}$ analysis stages, along with electronic and fluidic interconnects. Right: $\mu\text{ChemLab}^{\text{TM}}$ system packaged in a hand-held unit.

This monolithic construction has the advantage of handling all of the high frequency signals, providing DC-on, DC-off signals, in addition to reducing the size of the detection stage. The μ ChemLab™ components are mounted on a printed circuit board that makes both electrical and gas interconnects. Fig. 8 (left) shows a 5.3 x 8.5 cm board containing two complete analysis systems. This allows parallel analyses of analytes using different stationary phases on the two columns. Parallel analysis provides two different retention times for each analyte, thus increasing the resolution of the system. Supporting electronics required for temperature control of the components and sequential operation of the system is included on a separate PC board. The μ ChemLab™ system is packaged as a hand-held unit as shown in Fig. 8 (right).

References:

μ ChemLab Technology Team, "Autonomous Micro-Chemical Analysis Laboratory (μ ChemLab Technologies)," Sandia Report, SAND2001-1997, Sandia National Laboratories, Albuquerque, NM, Printed July 2001.

1. D. S. Ballentine, R. M. White, S. J. Martin, A. J. Ricco, E. T. Zellers, G. C. Frye, H. Wohltjen, Acoustic Wave Sensors: Theory, Design, and Physico-Chemical Applications, Academic Press 1997.

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